MEMORANDUM

TO: Mr. Addison Rice

Anderson, Mulholland and Associates

DATE: June 28, 2016

FROM: R. Infante

FILE: 1605308B

RE:

Data Validation
Air samples

SDG: 1605308B

SUMMARY

Full validation was performed on the data for several gas samples analyzed for methanol by Compendium Method TO-15. Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999". The samples were collected at the Bristol Myer Squib facility, Humacao, PR site on May 14, 2016 and submitted to Eurofins Air Toxics, Inc. of Folson, California that analyzed and reported the results under delivery groups (SDG) 1605308B.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B7IA-1 D	1605308B-02A	05/14/2016	Air	Methanol
B7IA-3	1605308B-04A	05/14/2016	Air	Methanol
B7IA-4	1605308B-05A	05/14/2016	Air	Methanol
B7IA-5	1605308B-07A	05/14/2016	Air	Methanol

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- o Initial and continuing calibrations

- o Method blanks/trip blanks/field blank
- o Canister cleaning certification criteria
- Surrogate spike recovery
- o Internal standard performance and retention times
- o Field duplicate results
- o Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- o Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form except for the following:

 The Chain of Custody (COC) information for sample B7IA-1D did not match the information on the canister with regard to canister identification. The client was notified of the discrepancy and the information on the canister was used to process and report the sample.

Holding Times and Sample Preservation

All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.

Initial and Continuing Calibrations

Methanol by Compendium Method TO-15

One point calibration performed. Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

No trip/field blank analyzed with this data package.

Laboratory/Field Duplicate Results

No field/laboratory duplicates were analyzed as part of this data set.

LCS/LCSD Results

Methanol

No LCS/LCSD (blank spike) was analyzed by the laboratory associated with this data package.

Quantitation Limits and Sample Results

Dilutions were performed (see worksheet).

Menda

LIC #

Calculations were spot checked.

Certification

The following samples 1605308B-02A; 1605308B-04A; 1605308B-05A; and 1605308B-06A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.

Rafael Infante

Chemist License 1888



Client Sample ID: B7IA-1D Lab ID#: 1605308B-02A

EPA METHOD TO-15 GC/MS

File Name: Dil. Factor:	14052009 1.66	Date of Collection: 5/14/16 8:12:00 PM Date of Analysis: 5/20/16 12:41 PM		
Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)
Methanol	170	Not Detected	220	Not Detected

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	104	70-130
Toluene-d8	101	70-130
4-Bromofluorobenzene	100	70-130

Pafael Infans Méndez



Client Sample ID: B7IA-3 Lab ID#: 1605308B-04A

EPA METHOD TO-15 GC/MS

File Name:	14052010	Date of Collection: 5/14/16 8:34:00 P			
Dil. Factor:	1.80	Date of Analysis: 5/20/16 01:42 PM			
Compound	Rpt. Limit	Amount	Rpt. Limit	Amount	
	(ppbv)	(ppbv)	(ug/m3)	(ug/m3)	
Methanol	180	Not Detected	240	Not Detected	

Container Type: 6 Liter Summa Canister (100% Certified)

		Method
Surrogates	%Recovery	Limits
1,2-Dichloroethane-d4	107	70-130
Toluene-d8	102	70-130
4-Bromofluorobenzene	98	70-130





Client Sample ID: B7IA-4 Lab ID#: 1605308B-05A

EPA METHOD TO-15 GC/MS

File Name:	14052011	Date of Collection: 5/14/16 8:57:00 PM		
Dil. Factor:	1.62	Date of Analysis: 5/20/16 02:09 PM		
Compound	Rpt. Limit	Amount	Rpt. Limit	Amount
	(ppbv)	(ppbv)	(ug/m3)	(ug/m3)

Methanol 160 Not Detected 210 Not Detected

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	105	70-130
Toluene-d8	101	70-130
4-Bromofluorobenzene	99	70-130



Client Sample ID: B7IA-5 Lab ID#: 1605308B-06A

EPA METHOD TO-15 GC/MS

File Name: Dil. Factor:	14052012 1.58	Date of Collection: 5/14/16 8:04:00 PM Date of Analysis: 5/20/16 02:29 PM					
Compound	Rpt. Limit (ppby)	Amount (ppby)	Rpt. Limit (ug/m3)	Amount (ug/m3)			
Methanol	160	Not Detected	210	Not Detected			

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method
Juliogales	MRECOVERY	Limits
1,2-Dichloroethane-d4	108	70-130
Toluene-d8	103	70-130
4-Bromofluorobenzene	99	70-130



eurofins

Sample Transportation Notice

Relinquishing signature on this document indicates that sample is being shipped in compliance with 180 BLUE RAVINE ROAD, SUITE B all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend,

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and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples, D.O.T. Hotline (800) 467-4922 Project Manager Terry lavin Project Info: Turn Around Lab Use Only. Time: Pressurized by: Collected by: (Print and Sign) _ Terry Taylor + Dayr) □ Normal Dater* Rush Project # Pressurization Gas: Phone 914-251-0400 Project Name N₂ He 3 4 Canister Pressure/Vacuum Date Time Lab I.D. Field Sample I.D. (Location) Can # of Collection of Collection **Analyses Requested** Receipt. 10-15 On 300 2011 2012 7.0 2025 13.0 To-15 10.0 2 004 +30 KK 00316 +30 10-15 511716 Relinquished by: (signature) Date/Time Received by: (signature) Date/Time Notes: 05/16/16 1500 Fed Relinquished by: (signature) Date/Time Received by: (signature) Date/Time Relinquished by: (signature) Date/Time Air Bilk# Temp (°C) Condition **Custody Seals Intact?** Work Order # Use Yes None 1675378 Only

	Project Number:1605308B Date:05/14/2016
REVIEW OF VOLATILE ORGANIC PACE The following guidelines for evaluating volatile organics were creat actions. This document will assist the reviewer in using professional decision and in better serving the needs of the data users. The sample USEPA data validation guidance documents in the following order "Compendium Method TO-15. Determination of Volatile Organic Com Specially-Prepared Canisters and Analyzed By Gas Chromatogra January, 1999"; USEPA Hazardous Waste Support Branch. Valida Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31 QC criteria and data validation actions listed on the data review works! document, unless otherwise noted. The hardcopied (laboratory name) _EurofinsAir_Toxics reviewed and the quality control and performance data summarized. The	led to delineate required validation I judgment to make more informed results were assessed according to of precedence: QC criteria from appounds (VOCs) In Air Collected In aphy/Mass Spectrometry (GC/MS), ting Air Samples. Volatile Organic I. Revision #4. October, 2006). The heets are from the primary guidance data package received has been
Lab. Project/SDG No.:1605308B No. of Samples:4	Sample matrix:Air
X Holding Times X X GC/MS Tuning X X Internal Standard Performance X X Blanks X	
Definition of Qualifiers: J- Estimated results U- Compound not detected R- Rejected data UJ- Estimated nondetect Reviewer: Date: 06/28/2016	

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
0.	_	
		
		- Yı
	- 1	
	-	
-	***	
		1
		N.
		- FA

All criteria were met _	Х_	_
Criteria were not met		
end/or see below		

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pН	ACTION
100				
All samples ana	lyzed within the recomi	mended method holding	time. Al	I summa canisters received
				B7IA-1D did not match the
				client was notified of the
IIIVIIIIUUVII VII I	uic cailistel milli icadi	u to cariistei luciiliite	UULI. IIIC	2 CIICIIL WAS IIVUIICA OI UIC
		canister was used to pr		

Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH \leq 2, 4°C), no air

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles. Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 ± 2 °C): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ) If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

All	criteria were met _	X
Criteria were	not met see below	

DATA REVIEW WORKSHEETS

GC/MS TUNING

The assessment of standard tuning Q		determine if the sample instrum	entation is within the
XThe BFB p	performance results were r	eviewed and found to be within th	e specified criteria.
_XBFB tunin	g was performed for every	24 hours of sample analysis.	
If no, use profess qualified or rejecte		ne whether the associated data	should be accepted,
List	the	samples	affected:
If mass calibration	is in error, all associated o	lata are rejected.	

All criteria were met _	X_
Criteria were not met	
and/or see below	

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	_05/20/2016
Dates of continuing calibration:	_05/20/2016
Instrument ID numbers:MSD	-14
Matrix/Level:Air	/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
			l and continuing calibra neet method specific re		pecific requirements. Initial

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be \leq 15 % regardless of method requirements for CCC.

All %Ds must be < 30% regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of \geq 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and nondetects (UJ).

If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r < 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were met	X
Criteria were not met	
and/or see below	

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method				
Field <u>/</u> Equipmen	t/Trip blank			
DATE ANALYZED	LAB ID	LEVEL <i>i</i> MATRIX	COMPOUND	CONCENTRATION UNITS
			n_this_data_package	
			73.00	
			<u> </u>	
				V 300-0-0-0-0-0-0-0-0-0-0-0-0-0-0-0-0-0-0

All criteria were met _X_	_
Criteria were not met	
and/or see below	

VB. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \le AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
					1000
		_			- CO
				-	
	155 AGE				

All criteria were met _X
Criteria were not met
and/or see below

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

SAMPLE ID

SURROGATE COMPOUND

d8

ACTION

1,2-DICHLOROETHANEd4 Toluene- 4-

4-BFB

Surrogate_recove	eries_within_laboratory	_control_limits		
QC Limits* (Air)				
li to til	70 to 120	70 to 120	70 to 120	

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

Actions:

_	QUALITY	%R < 10%	%R = 10% - LL	%R > UL
	Positive results	J	J	J
	Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met
Criteria were not mel
and/or see belowN/A

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

	PD of the compounds		not meet the criteria. Matrix/Level:			
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION	
MS/MSD_accuracy_	_are_not_required_as	_part_of_l	Method_	TO-15;_blank_sp	ike_used_to_assess_	

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

^{*} QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

^{*} If QC limits are not available, use limits of 70 – 130 %.

All criteria were met _____ Criteria were not met and/or see below __N/A___

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:			Matrix/Level/Unit:			
COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION	
			20	10 to		
			-	1	<u> </u>	
			E 100			
V						
	700					
Contract of the Contract of th	Ω.					

Actions:

^{*} If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

^{*} If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were met _	х_
Criteria were not met	
and/or see below	

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

LOCID

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

	LC2 ID	COMPOUND	% R	QC LIMIT
No_LCS	/LCSD_(Blank_s	spike)_analyzed_in_this_da	ata_package	

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or <u>No</u>. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

		All criteria were metN/A Criteria were not met and/or see below
IX.	LABORATORY DUPLICATE PRECISION	1
	Sample IDs:	Matrix:

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD ± 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION	
o field/labora	tory duplicate	analyzed with	this data	nackage	
		diayzed war	JIIS GAILE	paokago.	
		CONC.	CONC. CONC.	CONC. CONC.	

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were metX
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within \pm 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
	tandard_area_and_re ation_standards	etention_times_	within_laboratory	_control_limits_for_	both_samples
Actions:					

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%	IS AREA > + 40%		
Positive results	J	J		
Nondetected results	R	ACCEPT		

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were met _X__ Criteria were not met and/or see below ____

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1605308B-02A

1,2-Dichloroethane-d4 RF = 2.06681

[] = (304177)(400)/(140977)(2.06681)

= 417.6 ppbv OK

All criteria were met_	X_
Criteria were not met	
and/or see below	_

XII. QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
All samples dil	uted by a factor of less th	an 1.8.
		SECTION SECTION
	7000	
15000		

B.	Percent	Solids
----	---------	--------

List samples whi	ch have ≤ t	50 % soli	ds				
					11:		W.
			GE E	45000			
THE REAL PROPERTY.	P. S. S. A.	122					

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R) $\,$